⑫ EUROPEAN PATENT APPLICATION

㉝ Optical analysis method and apparatus having programmable rapid random wavelength access.

㉗ The optical reflectance or transmittance concentration analyzer includes an acousto-optical tunable filter (AOTF) with a pair of crossed polarizers (3, 5) to select one of the tuned monochromatic light beams that pass from a light source (1) through the AOTF. The AOTF is tuned by a computer controlled digital-to-analog converter (12) through a tunable sweep oscillator (11). To obtain rapid wavelength change along with electronic chopping or wavelength modulation, the digital-to-analog converter output is added to the output of a high speed signal generator (18). The modulated light is conducted directly or through a fiber optic cable to the location of the optical measurement and impinges upon the sample where it is reflected or transmitted. The emerging light is collected onto detector(s) (10). The measurement time interval can be distributed among the required wavelengths or wavelength pairs according to an unequal scheme to decrease the error of the result in a calibration equation. The apparatus is particularly adaptable for use as an on-line concentration monitor in industrial process control because of tis stability, efficiency and ability to perform quick analyses.

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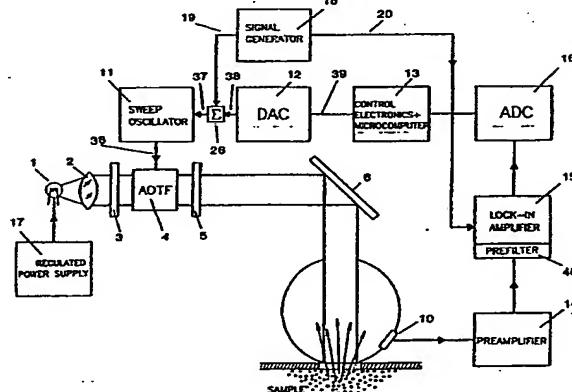
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OPTICAL ANALYSIS METHOD AND APPARATUS HAVING PROGRAMMABLE RAPID RANDOM WAVELENGTH ACCESS

BACKGROUND OF THE INVENTION

1. FIELD OF THE INVENTION

5   The present invention relates to optical analyzing instruments, and more particularly to reflective or transmissive optical analyzing instruments for determining the composition of solid, liquid, slurry or paste samples by their near-infrared, infrared or

10   visible absorbances, especially for industrial on-line monitoring applications.

2. DESCRIPTION OF THE PRIOR ART

      An empirical correlation near-infrared reflectance analysis method was first suggested and

15   elaborated on by Karl H. Norris of the United States Department of Agriculture, Beltsville, Maryland, in the mid 1960s. It was observed that different wavelengths of near-infrared radiation incident on the surface of the sample are absorbed or reflected to certain extents,

20   depending on the characteristics and concentrations of the constituents of the sample. The reflected radiation is collected by suitable optics and measured by a suitable detector arrangement. The concentrations of the material to be measured can be calculated from the radiation

25   intensities measured at different wavelengths.
      A more detailed history and background on this subject is contained in the article "Near-Infrared Reflectance Analysis" by David L. Wetzel, Analytical Chemistry, vol. 55, p. 1165A (American Chemical Society

30   1983).

Various instruments comprising different monochromators have been previously described for accomplishing the above results. Most of these systems were directed to laboratory analysis of samples.

5 However, instrumental principles and systems disclosed in the prior art are not practical to use in industrial on-line concentration monitoring.

For example, in on-line monitoring, the samples are moving and, in many cases, are inhomogeneous. Thus,

10 to achieve the monitoring goals, a large number of measurements must be made in a very short time and the results averaged to reduce errors. In addition to the averaging requirement, it is necessary to correct for the rapid changing of the sample. It is also desirable to

15 use optical data taken from the same part of the sample in a calibration equation. These results can be achieved in principle by applying a "stopped-flow" sampling, but this procedure is less representative of the bulk of the sample and is also much too slow for most control

20 purposes.

Prior art interference filter systems with a perpendicular direction of light beam are described in U.S. Patents 3,828,173 and 4,236,076. In both systems, discrete wavelength interference filters are mounted in a

25 turret. Thus, the rate of the wavelength selection is limited by the mechanical means to rotate the turret.

Of special note are interference filter instrument systems described in U.S. Patents 4,286,327 and 4,404,642. As light sources, infrared emitting

30 diodes (IREDs) are used, whereas all other known instruments in the prior art utilize wide wavelength band quartz tungsten-halogen light sources. The advantage of the special light source is that it has a relatively low dissipation in comparison with conventional light sources

35 and can be activated very rapidly by a timer through a microcomputer. In exchange for these advantages, the wavelength region is constrained to the region of the

infrared emitting diodes (about 850-1050 nm). In this
constrained region, a very sensitive Si detector can be
used but only a few chemical components show
characteristic absorbances.

5   Tilting interference filter systems are described
in U.S. Patents 3,861,788 and 4,082,464. The
interference filters are mounted on paddle wheels, and
rotated to provide a wavelength shift as the angle of the
filter and the incident beam varies in time. These
10 systems produce continuous wavelength change, but only
very small fractions of the whole rotation can be
considered "useful" time, when the filter is accurately
producing the required wavelengths. There is also
considerable "dead" time when the beam is mechanically
15 blocked between filters.

Diffraction grating systems for optically
analyzing samples are also known. For example, vibrating
holographic grating systems, capable of up to ten scans
per second are shown in U.S. Patents 4,264,205,
20 4,285,596 and recently in U.S. Patent 4,540,282. The
vibrating grating principle allows only sequential access
to the individual wavelengths as the whole spectrum is
swept through in time. One of the disadvantages in the
high speed applications of grating monochromators is that
25 the signal-to-noise ratio cannot be enhanced by chopping
and narrow noise bandwidth phase sensitive (lock-in)
amplification. This technique can only be applied in
slow point-to-point scan grating systems, where the
grating is stopped at every required wavelength, and
30 enough light chopping periods are allowed to elapse for
the signal intensity to be precisely measured. Thus, the
total spectrum measurement time is increased up to about
a minute.

Another trade-off in mechanical (rotating sector)
35 chopper systems is, that only half of the measurement
time is utilized by the detector for the purpose of
collecting light at the required wavelength. In the
other half of the period, a fast dark compensation

occurs, carrying no "wavelength information". Lock-in amplification is described in U.S. Patent 4,236,076, where the light is modulated by chopping the light, periodically referencing by a tilting mirror and changing

5   the wavelength by changing interference filters in the beam. A disadvantage of this system for the monitoring of rapidly changing material is that the wavelength change is the slowest of the mentioned three modulations, the rate being several seconds between consecutive

10   wavelength choices.

Further increases in the speed and improvement of the efficiency of the monochromator and the analyzer system lead to serious problems with mechanical monochromatic systems that are known in the art.

15   Non-mechanically tunable optical devices including acousto-optical tunable filters (AOTF) have been described in the article "Acousto-Optic Tunable Filter" by S. E. Harris and R. W. Wallace, Journal of the Optical Society of America, vol. 59, pp. 744-747 (Optical

20   Society of America 1969). An early described model was tunable from 400 nm to 700 nm by applying 428-990 MHz acoustic frequency via an acoustic transducer layer attached to a LiNbO crystal. Since then, various acousto-optic devices have been described in

25   "Noncollinear Acousto-optic Filter with Large Angular Aperture" by I. C. Chang, Applied Physics Letters, vol. 25, pp. 370-372 (American Institute of Physics 1974), and also disclosed in U.S. Patents 3,679,288; 3,944,334; 3,944,335; 3,953,107; 4,052,121, and 4,342,502. In the

30   above-mentioned prior art, the geometry, the material of the crystal used and the optical arrangement varies.

However, one common feature is that all acousto-optic tunable filters utilize the principle that the direction of propagation and the direction of

35   polarization of an appropriate incident ray is changed by the application of a high frequency optical modulation of the crystal. The different frequencies give rise to different densities of index of refraction fronts due to

local stresses caused by the acoustic waves. This tuning
can be activated over a relatively wide frequency
(wavelength) range, thus rendering the device ideal for
optical tuning purposes.

OBJECTS AND SUMMARY OF THE INVENTION

An object of this invention is to improve the
speed of the wavelength change to increase scan rate for
better averaging of moving or changing samples.

Another objective of the present invention is to
improve the efficiency of the near-infrared optical
analysis using a measurement scheme, where the unused
optical instrument time is minimized.

Yet another objective is to reduce the thermal
and vibration sensitivity of the presently known optical
analyzers to permit their application to industrial and
process conditions.

The present invention is an improvement over
prior optical reflectance or transmittance analyzers.
The improvements make the analyzer faster, more
efficient, more stable and mechanically less complicated,
thus also smaller and lighter. The above characteristics
render the instrument able to be used as an on-line
concentration monitor in industrial process control.

One of the basic recognitions in the present
invention is that the increase of the wavelength
selection rate has substantial limitations if pursued in
a mechanical manner. It was therefore established that
one of the electro-optical, electromagneto-optical or
acousto-optical tunable devices must be included in a new
optical system in order to achieve shorter wavelength
access time. On the basis of the wavelength range and
tunability, the AOTF was preferred for incorporation in a
new system.

It should be noted that the optimal wavelengths
and wavelength combinations vary from application to
application. Thus, in a general purpose instrument it is

very desirable to have access to all wavelengths in any
random manner.

It was further recognized that random wavelength
access is desirable and possible with an AOTF while
avoiding the unnecessary "dead" times typically
associated with rotating or tilting filters, or the
serial wavelength accessibility typical of the grating
monochromator systems.

It was also recognized that along with the
computer controlled electronic wavelength selection, the
necessary chopping or wavelength modulation can also be
carried out without any moving parts, thus significantly
simplifying the construction of such an optical analysis
instrument.

It was further recognized in the present
invention that with the previous "on-off" chopping mode,
every second half-cycle of time carries no wavelength
characteristic information. When the radiation is
blocked, the signal is used only to correct for the
background.

With a fast tunable device, as in the proposed
AOTF wavelength modulation, wherein the radiation is not
blocked in every half cycle, the signal intensity is
measured at an alternative wavelength. The frequency of
this wavelength modulation is used as a reference for a
phase sensitive amplification.

It is also recognized that tuning can be carried
out by initiating the change of the base wavelength
selection, providing a constant periodic signal during
the whole measurement.

It is further recognized that complex optical
instruments containing numerous moving parts are more
susceptible to failure, more sensitive to vibration and
to changing thermal conditions. In addition, the
instruments in the prior art containing interference
filters are sensitive to thermal changes, resulting in a
shift of the peak transmittance wavelength with different
temperatures. The simple optical layout and the nature

of the wavelength selection of the AOTFs according to the present invention are insensitive to vibrations or to ambient temperature changes except to sudden temperature shocks.

5   The objects of the invention are achieved by a system comprising an AOTF. The light emerging from a wide band source, such as a quartz tungsten-halogen lamp, is collimated and polarized by a suitable polarizer. Relatively inexpensive sheet-type polarizers are

10   available. However, their extinction ratio is not uniformly good throughout the entire visible or near-infrared spectrum. Glan-Thompson or Glan-Taylor type polarizers develop an extinction ratio better than 10^{-5}, thereby supressing the non-modulated light much better,

15   but are more expensive. The input and output beams are colinear as in the case of the sheet polarizers. Wollaston prism type polarizers are also potentially useful because of their good extinction ratio, but the input and output beams are not colinear, and the angle

20   deviation must be considered in the optical design. With crossed polarizers placed in front of the AOTF and behind it, only the tuned ordinary or the tuned extraordinary beam is selected and the unmodulated traversing light is diverted or absorbed by the second

25   polarizer. As the input polarization direction, either the vertical or horizontal direction can be chosen. The selection determines which of the output beams can be used.

        The AOTF is driven by a sweep oscillator, which

30   provides the necessary input for the acoustic transducer mounted on the crystal. For easy control interfacing, the voltage tuned sweep oscillators are advantageous.

        It was also recognized that in order to utilize the advantages of a high speed system, the choice of

35   detectors should be based on their speed along with their sensitivity, noise equivalent power (NEP) and wavelength range. In contrast to most other optical analyzers in the prior art, where Si or PbS detectors are used, the

apparatus according to the present invention along with the Si, PbS, PbSe detectors may comprise Ge or cooled Ge detectors to match the speed of the AOTF and yet retain a wide enough wavelength range.

5   A further recognition is that the fiber optic light transmission can very advantageously be applied in conjunction with the AOTF. Optical fibers are small diameter light conducting materials, with a small light input diameter and a limited light acceptance cone. In 10 order to couple light into optical fibers, the light must be focused onto the end of the small fiber with no more than the cone of acceptance. The non-distorted, collimated (and modulated) light emerging from the AOTF is ideally suited for focusing with the above 15 requirements, with the help of a simple aspheric lens or an off-axis parabolic mirror.

  The result of the optical analysis is calculated by substituting the measured light intensity values into optimal calibration equations.

20   **DESCRIPTION OF THE DRAWINGS**

  In the accompanying drawings,

  FIG. 1 shows an optical arrangement of a commercially available AOTF and the polarization characteristics thereof;

25   FIG. 2 shows a near-infrared spectral portion (reflectance spectra) of wheat samples with different (increasing) moisture content;

  FIG. 3 shows two tuning control schemes for a tunable sweep oscillator incorporating the present 30 invention;

  FIG. 4 shows different tuning schemes for the tunable sweep oscillator input;

  FIG. 5 shows the optical path and basic components of the apparatus incorporating the invention;

35   FIG. 6 shows a simplified schematic electronic block diagram thereof;

FIG. 7 shows a fiber optic transmission of
modulated light to the site of the measurement; and,

FIG. 8 shows a simplified schematic block diagram
of a remote optical head utilizing fiber optic illumination.

DETAILED DESCRIPTION OF THE INVENTION

Referring to Fig. 1, if the wide band input
light is polarized, another polarizer (the so-called
analyzer) can select the tuned monochromatic ray from
the traversing untuned polychromatic light.   In a
commercially available AOTF having an optical
arrangement such as shown in Fig. 1, the wavelength
accessibility is influenced by the size and geometry of
the acousto-optic crystal and the velocity of the sound
traveling in the crystal.   Thus, a 100 to 100,000-fold
advantage in the speed of the wavelength change rate may
be obtained compared to other prior art analyzers.   .

Referring to Fig. 2, a portion of the near
infrared reflectance spectra of six ground wheat samples
with increasing (different) moisture contents is shown.
As will be seen, this constituent can be quantitated by
measuring the height of the peak around 1940 nm.   To
correct for the baseline changes, most pronounced on
spectrum 6, a reference wavelength (λ_{ref}) can be used,
for example.   Another option is to use the difference
spectrum, the extreme case being the derivative of the
spectrum.   This approach will not contain the baseline
shifts originating from particle size differences,
background radiation or other effects, but only the
steeper spectral changes.   Thus, the differences between
the intensities between λ_1 and λ_2 also characterize
the amount of moisture in the sample.   Another or a
plurality of differences (λ_3, λ_4, etc.) can be
included in the calibration equation.

Tuning schemes, such as shown in Fig. 3, include
a selection of different wavelengths by applying
different control input voltages (U_c) to a tunable sweep

oscillator. Under this arrangement, there is either no lock-in amplification or only a mechanical chopping, which should be faster than the wavelength change. A control scheme such as shown in Fig. 3b, furnishes rapid

5 on-off tuning of the sweep oscillator. Besides selecting λ_1, λ_2 or λ_3 (by selecting U_c control voltages) the rapid on-off provides electronic chopping and does that only on the selected wavelength.

With a phase sensitive detection (lock-in

10 amplification), only the preferred selected wavelength information is amplified and all other background signals and noise of other frequencies are suppressed. With this chopping scheme, the frequency is controlled electronically and is thus kept very precise in

15 comparison with frequency error experienced with mechanical chopping devices.

The band width of the amplification is therefore much narrower, reducing the noise of the apparatus. The noise can be further reduced by the usage of a narrow

20 band pass prefilter or other electronic means.

Various possible tuning schemes are contemplated as shown in Fig. 4. Fig. 4a shows $\lambda_1 - \lambda_{ref} - \lambda_1 - \lambda_{ref} \cdots \lambda_2 - \lambda_{ref} - \lambda_2 - \lambda_{ref}$. sequences, where λ_{ref} is constant throughout the whole experiment. The

25 $\lambda_1 - \lambda_{ref}$ sequence is repeated as many times as required by the output of the lock-in amplifier (LIA) to be settled (typically 10-12 cycles) and sampled by the analog-to-digital converter (ADC), connected to the output of the lock-in amplifier. The lock-in amplifier

30 usually has very narrow bandpass characteristics, but optionally the signal-to-noise ratio (SNR) can be further enhanced by inserting a narrow band preamplifier at the input of the lock-in amplifier.

First derivative (first difference) spectrum

35 modulation scheme is shown in Fig. 4b. The difference of the consecutive wavelength pairs is kept constant ($\lambda_2 - \lambda_1 = \lambda_4 - \lambda_3$), and similarly each pair is repeated as long as one measurement point can be taken

and recorded via the lock-in amplifier and the ADC.

Tuning can be carried out by initiating the change of the base wavelength selection (λ_1 and λ_3) by a computer controlled digital-to-analog converter (DAC) via the tunable sweep oscillator, whereas the chopping modulation preferably originates from a signal generator circuit, providing a constant periodic signal during the whole measurement. This latter signal is simply added to the aforementioned base signal, the total control signal (U_c) being shown in Fig. 4b.

If only a few wavelengths are used for analytical purposes, they can be randomly selected by the computer control, as shown in Fig. 4c. The tuning difference is not kept constant in this instance.

Although any of the tuning schemes of Figs. 3 and 4 can be used, the scheme of Fig. 4b is easily implemented up to very high speeds without sophisticated computer hardware or software. A separate signal generator provides the high frequency (up to 100 kHz) square-wave or sinewave signal that is added to the much slower changing base wavelength selection signal (0-10 kHz) generated by the DAC circuit, controlled by a microcomputer.

The reference signal from the signal generator circuit serves as an input for the narrow band phase sensitive amplifier (LIA). It is preferable to permit approximately 10-12 cycles of time to elapse for the LIA output to settle. The output is filtered and introduced into a computer controlled ADC. The rate of data acquisition is the rate of the base wavelength change. Therefore, it can be about 10-12 times slower than the highest speed event, the primary periodic wavelength modulation.

The fiber optic transmission of light from the monochromator, in this case from the AOTF to the optical head, where the light comes in contact with the sample, is advantageous. Only some minor electronic and optical components are exposed to the process environment, and

the light source, monochromator, and most related
electronics and computing means can be located
elsewhere. The optical head serves to illuminate the
sample and collect the radiation emerging from the
5 sample.

If the sample is solid, the diffusely
transmitted or reflected light cannot be gathered with
sufficient efficiency back into another fiber. Thus,
two different optical approaches are pursued. For clear
10 liquids, the transmitted light may return to the main
optical/electronic unit for detection via a second
optical fiber, thus isolating the optical head
electrically. This feature may be very advantageous in
the determination of concentrations in explosive or
15 otherwise hostile environments.

If the sample is diffusely scattering the light,
it should be collected by an integrating sphere or a
suitable mirror directly onto a (larger surface)
detector. In this case, the optical head contains the
20 collection optics, the detector(s) and the detector
related electronics.

The result of the optical analysis is calculated
by substituting the measured light intensity values into
optimum calibration equations. It should be noted that
25 the error of the analysis depends upon the error of the
individual optical measurement and on the magnitude of
the constant with which it is multiplied. The errors
contributed to the individual terms are shown in the
following equation 1.

30 $$Y[\%] = K_0 + K_1(I_1 + \varepsilon_1) + K_2(I_2 + \varepsilon_2) + K_3(I_3 + \varepsilon_3) .. \quad (1)$$

where Y is the chemical constituent to be measured, K_0
through K_3 are constants, I_1 is the signal intensity at
wavelength λ_1, and ε_1 is the error at $_1$.

In order to reduce the total measurement error,
35 the sum of the products $K_1\varepsilon_1$ must be decreased. With
the computer controlled electronic tuning, this error

can be decreased by allowing different measurement times
and number of averages for the different wavelengths.

It is known that by increasing the measurement
time or the number of averages by N, the signal-to-noise
ratio can be increased by N. In a certain calibration
equation the given errors are multiplied by widely
varying constants. Therefore, it is desirable to
decrease the error at the wavelength where the K
constant is large.

It is cumbersome to derive an analytical
equation for optimum division of time between the
various wavelengths, mainly because of the square root
function of the error reduction. However it can be seen
that for every error-constant distribution, there is an
optimum time distribution, other than the equal sharing
of measurement time.

In order to decrease the error, a longer time
must be spent at wavelengths with larger constant
multipliers. If we increase the time at one wavelength
after a certain point, some other error terms start
increasing at the remaining wavelength with the shrinking
time compromising even the terms with smaller constants.

Fig. 5 shows the optical path of one of the
preferred embodiments wherein a light source 1 is an
incandescent wide-wavelength band quartz tungsten-
halogen lamp. The light emerging from the light source
1 is collimated by a collimating lens 2.

The preferred embodiment is not limited to lens
type collimating devices. Collimating mirrors,
preferably off-axis parabolic mirrors, can also be used.

The collimated light traverses through a
polarizer 3, which selects one of the polarization
components and either absorbs, reflects or directs the
other component elsewhere. The collimated and polarized
light enters an acousto-optic tunable filter (AOTF) 4.
This AOTF 4 is arranged in a position and angle for
optimum acceptance of the polarized input light for
subsequent modulation.

Modulated monochromatic and unmodulated light emerges from the AOTF 4. The unmodulated component retains its angle of polarization, whereas the modulated component, namely the light with the selected wavelength,

5   alters its direction of polarization by 90 degrees. The polarizer 5 is in a crossed position relative to a polarizer 3. Thus, only the selected modulated light component traverses through the polarizer 5.

The polarizers can be of any suitable known type

10   that is effective in the selected wavelength region. However, the second polarizer 5 should be sensitive to small angle deviations of the emerging tuned beam. The sheet polarizer, for example, should be arranged perpendicular to the emerging tuned beam. The angle

15   deviation may vary from 1 to 10, depending on the arrangement of the crystal (not shown) used in the AOTF 4 and the choice of the ordinary or the extraordinary beam. However, this small angle deviation is not shown in Fig. 5.

The selected modulated wavelength beam is

20   directed onto the surface of the sample. In one preferred embodiment this is done by a mirror 6, through an optical window 8. A sample 9, in this case solid material, is placed near the opening of an integrating sphere 7. The light impinging on the sample 9 is partly

25   reflected and partially penetrates the sample 9 where it is subjected to scattering, reflection and absorption. As a result, light emerges from the sample 9 back into the integrating sphere 7. The light encounters multiple reflections and eventually falls onto the surface of a

30   detector 10.

The detector 10 can be one or more of the following types: Si, PbS, PbSe, Ge or cooled Ge, to enhance the long wavelength limit of this type of detector.

35   Referring to Fig. 6, the simplified electrical block diagram of the preferred embodiment, shows a sweep oscillator 11 connected to the AOTF 4, preferably via a short coaxial cable 36. The sweep oscillator 11

generates the necessary RF signal to drive the AOTF 4.
Depending upon the design of the crystal (not shown) used
in the AOTF 4 and the required wavelength range, the
acoustic frequency is several MHz to several hundred MHz.

5   The output power at an adjusted impedance, to be
coupled into the crystal (not shown) used in the AOTF 4,
should be sufficient to drive the crystal into saturation
and provide maximum modulation efficiency. In the
preferred embodiment the sweep oscillator 11 is tuned to

10   different output frequencies by a high impedence analog
input. An analog control input 37 can be provided by a
preset or controlled analog voltage source. In this
embodiment a controlled voltage analog output 38 is
ensured by a digital-to-analog converter (DAC) 12, which

15   is driven by a microcomputer controller 13 through a
standard computer communication line 39.

          The signal from the detector 10 is introduced
into a preamplifier 14 and the output of the
preamplifier 14 is connected to the signal output of a

20   lock-in amplifier (LIA) 15. An optional prefilter 40
may enhance the signal of known modulation frequency to
suppress random noise of other frequencies. The LIA 15
can generate its own reference signal by forming the
amplified detector signal itself. An external reference

25   signal 20 is provided and connected to the reference
input of the LIA 15.

          A signal generator circuit 18 generates
sinusoidal or rectangular output, which is connected to
a summation circuit 21, and a corresponding reference

30   signal 20. Signals 19 and 38 are added to provide a
combined output 37 for wavelength selection and
wavelength modulation, respectively. A regulated power
supply 17 is connected to the light source 1 to provide
constant, noise-free light flux.

35   In one specific example of the preferred
embodiment, the light source is a 50 watt tungsten-
halogen lamp driven by a 5 volt DC collimating lens such
as an f-17 mm aspheric lens, as made by Melles Griot of

Irvine, California. The pair of polarizers can be iodine dyed polyvinyl-alcohol type sheet polarizers for the near-infrared region such as Model 27360 made by Oriel Corporation of Stratford, Connecticut. The acousto-optic tunable filter 4 is, for example, Model EFL-F20R3A made by Matsushita Electronic Components Company of Kadoma, Japan, with an optical aperture of 7 x 7 mm. The optical tuning range is 1.4 - 2.5 µm, with a deflection efficiency greater than 70 percent. The fastest wavelength access time is 11 µs. The optical resolution varies as a function of scan speed and the efficiency of the collimation of the input light.

The AOTF 4 is powered by, for example, a Model N-2117 sweep oscillator made by Interactive Radiation Inc. of Northvale, New Jersey. The tunable output of the sweep oscillator is selectable in the range of 34 - 63 MHz by analog input voltages between 0 - 6.5 volts. In this example, the sweep oscillator is controlled by, for example, a Model 59501B Isolated DAC/Power Supply Programmer made by Hewlett-Packard of Palo Alto, California, and an F34 Function Generator made by Interstate Electronics Corporation. The base wavelength is selected through, for example, an HPIB bus interface accessed from an HP 85A Microcomputer Controller made by Hewlett-Packard of Palo Alto, California.

For detection, lead sulfide detectors (10x10 mm) of the type sold by Infrared Industries Inc., were used at ambient temperature. The optical data was manipulated for composition analysis by the HP 85A microcomputer.

In another specific example of the preferred embodiment as shown in Fig. 7, the optical head was separated from the main instrument body by several feet. An instrument housing 41 contained a light source, the AOTF 4, the polarizers 3 and 5 and related electronics 42, with the additional focusing lens 22 (f=25 mm aspheric lens) as made by Melles Griot of Irvine, California, and the fiber optics 23.

The modulated light of the selected wavelength
is coupled by focusing lens 22 into fiber optic cable
23, capable of carrying the near-infrared radiation to a
remote test point, the maximum distance being determined
by the attenuation over the length of the fiber. At the
remote location of this example, such as at the site of
the chemical composition measurement for process control,
light irradiates the sample 9 directly or as shown in
Fig. 7 through the collimating lens 24. In the case of
solid samples, the reflected light is collected by an
integrating sphere onto the detector 10. The signal is
fed into the electronic processing signal unit 43.

In a further specific example, a simplified
schematic block diagram of the remote head of the
preferred embodiment is shown in Fig. 8. The remote
head contains the illuminating fiber optics 25 which is
shown without any further collimation or refocusing and
without collection optics. The reflection detectors 28
are mounted on temperature controlled blocks,
temperature controlled by a temperature regulator
circuit 30 powered through a supply connection 34. A
thermistor 44 provides the control signal for the
temperature regulator circuit 30.

In this example, the detectors used are lead
sulfide detectors biased to about 200 volts DC by a
regulated power supply 29, powered by line voltage 22 or
germanium detectors without further temperature control.
In yet another example, a silicon or germanium detector
28a is placed on the other side of a sample layer 27,
for transmittance measurement. The sample is
illuminated through an optical window 26, and the light
is measured in this latter example through an optical
window 26a. The small signal from the detectors is
amplified by preamplifier 31 and the amplified signal is
transmitted through a signal line 33 to the main
instrument electronics. It will be appreciated that the
simple optical head arrangement makes it possible to
couple more than one remote head to one central

instrument unit with the help of optical and electronic
multiplexing for multipoint process analytical
applications.

In any of the above examples, all the tuning
schemes of Figs. 3 and 4 can be used. In order to
minimize the error of the analysis, the timing of the
wavelength change (base wavelength change) need not be
equal.

CLAIMS

1. An apparatus for providing rapid random wavelength access for optical analysis of a sample comprising,

      (a) a light source,

      (b) an acousto-optic tunable filter positioned to accept light from the light source to provide emerging tuned output beams,

      (c) means to select a predetermined number of tuned beams from the acousto-optic tunable filter,

      (d) means to direct said predetermined number of selected tuned output beams onto the sample to be analyzed,

      (e) means to collect radiation that is reflected back or transmitted through said sample,

      (f) means for detecting and amplifying the reflected or transmitted radiation,

      (g) electronic tunable oscillator means connected to said acousto-optical tunable filter, and

      (h) electronic means providing timing and logic for wavelength selection via said electronic tunable oscillator means.

2. The apparatus of claim 1 further comprising optical focusing means and a fiber optic connection between said predetermined number of selected tuned output beams and said sample.

3. The apparatus of claim 2 further comprising a fiber optic coupling between said sample and said detection means.

4. The apparatus of claim 1 wherein said electronic tunable oscillator is controlled by a computer controlled digital-to-analog converter.

5. The apparatus of claim 4 further comprising square or sine-wave generator means and phase sensitive (lock-in) amplifier means.

6. The apparatus of claim 5 wherein the electronic tunable oscillator means has an oscillator output and the computer controlled digital-to-analog converter has a digital-to-analog converter output and the output of said square or sine-wave generator means is coupled into the electronic tunable oscillator means such that the oscillator output is added to the control signal output of the digital-to-analog converter output.

7. The apparatus of claim 1 wherein the detection means is formed from at least one material selected from the group consisting of germanium, lead sulfide, lead selenide or silicon.

8. The apparatus of claim 1 wherein the detecting means communicates with a prefilter and said detecting means furnishes a detector signal to said prefilter before said detector signal is amplified.

9. The apparatus of claim 1 further comprising means to collimate light emerging from said light source.

10. The apparatus of claim 9 further comprising a first polarizing means positioned to select at least one polarization direction from said collimated light.

11. The apparatus of claim 10 further comprising a second polarizing means positioned to select one of the emerging tuned output beams.

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12. A method of optical concentration and analysis of a sample wherein monochromatic light of different wavelength is selected in rapid succession with an acousto-optic tunable filter, the successive wavelengths of monochromatic light are guided onto the sample, the reflected or transmitted light intensity is measured and the concentration of the sample is determined by substituting the intensities at different wavelengths or intensity differences at various wavelengths into a calibration equation.

13. The method of claim 12 wherein the wavelength modulation is such that each wavelength or wavelength pair is selected for different time intervals.

Acousto-optic
crystal

Unpolarized input

α_1 α_2 α_3

A

B

C

RF input
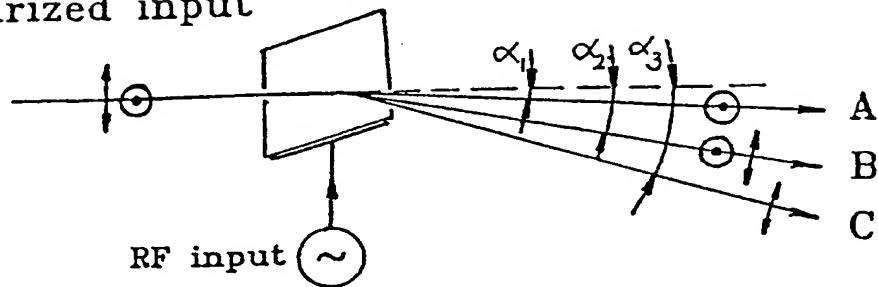
Horizontal polarized input

Polarizer 1

Polarizer 2

A

RF input
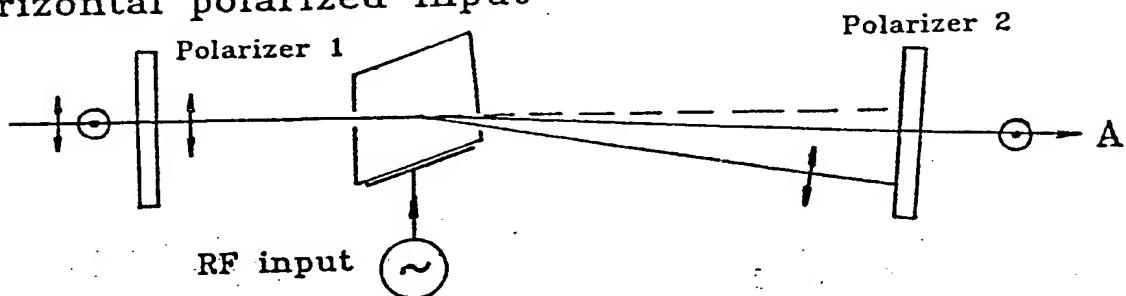
Vertical polarized input

Polarizer 1

Polarizer 2

C

RF input
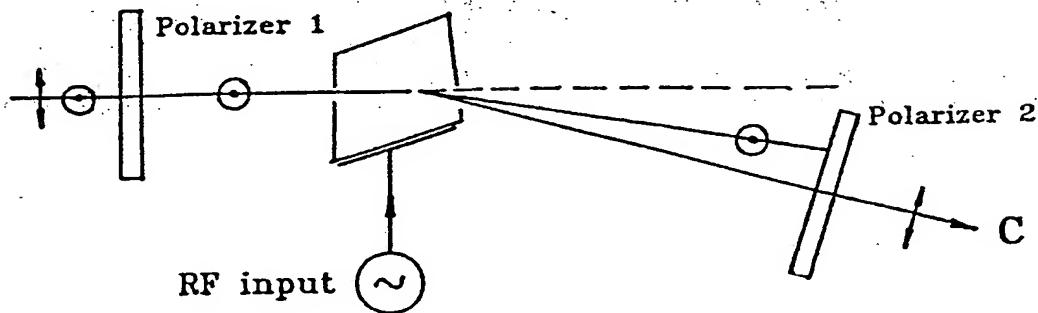
A. Tuned extraordinary light
B. 0th light (transmitted)
C. Tuned ordinary light

Fig 1

Fig. 2

Fig. 3

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Fig. 4

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Fig. 5

SAMPLE

Fig. 6

Fig. 7

TUNING ELECTRONICS

42

AOTF

22

23

24

1

10

9

SIGNAL PROCESSING ELECTRONICS

43
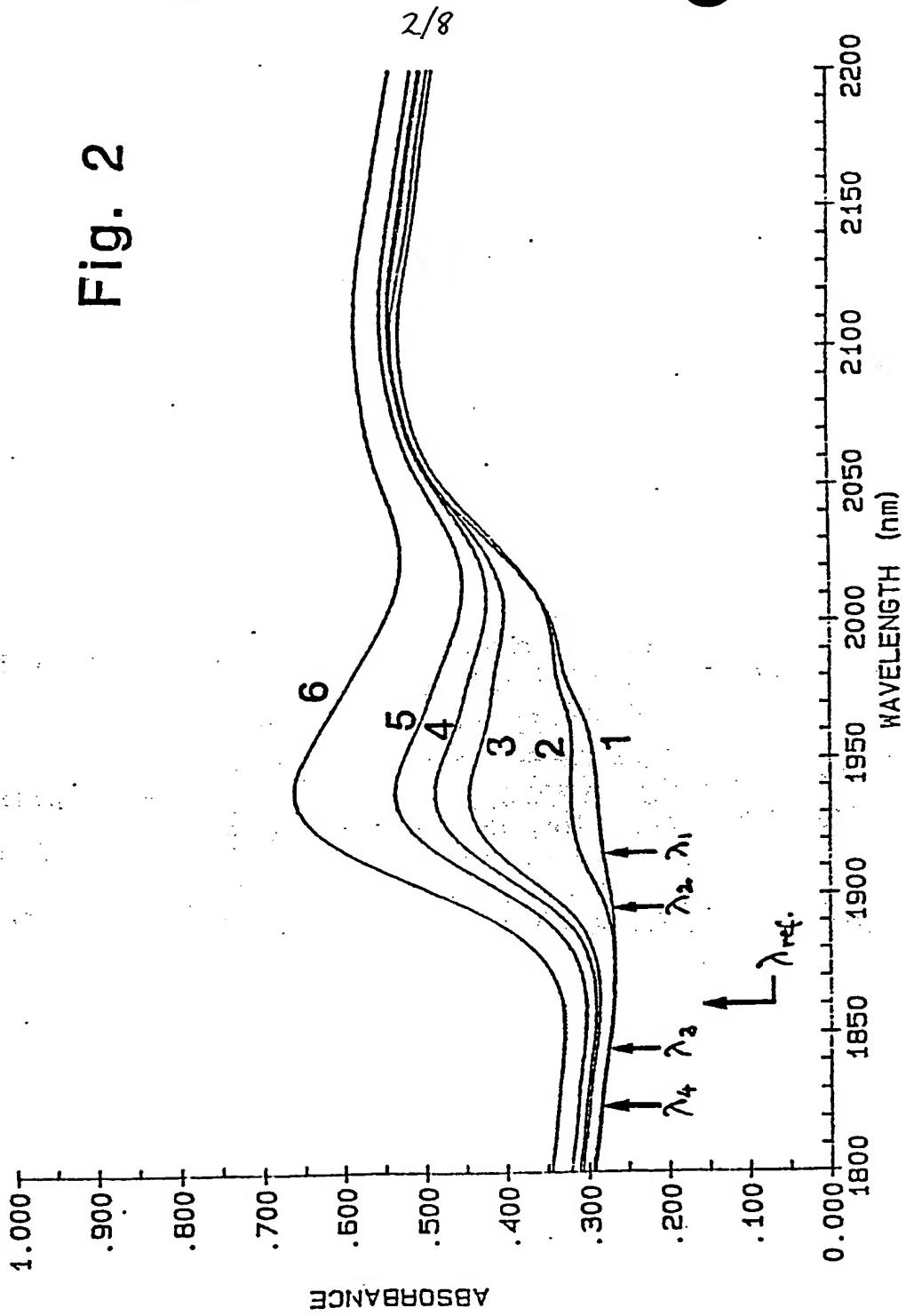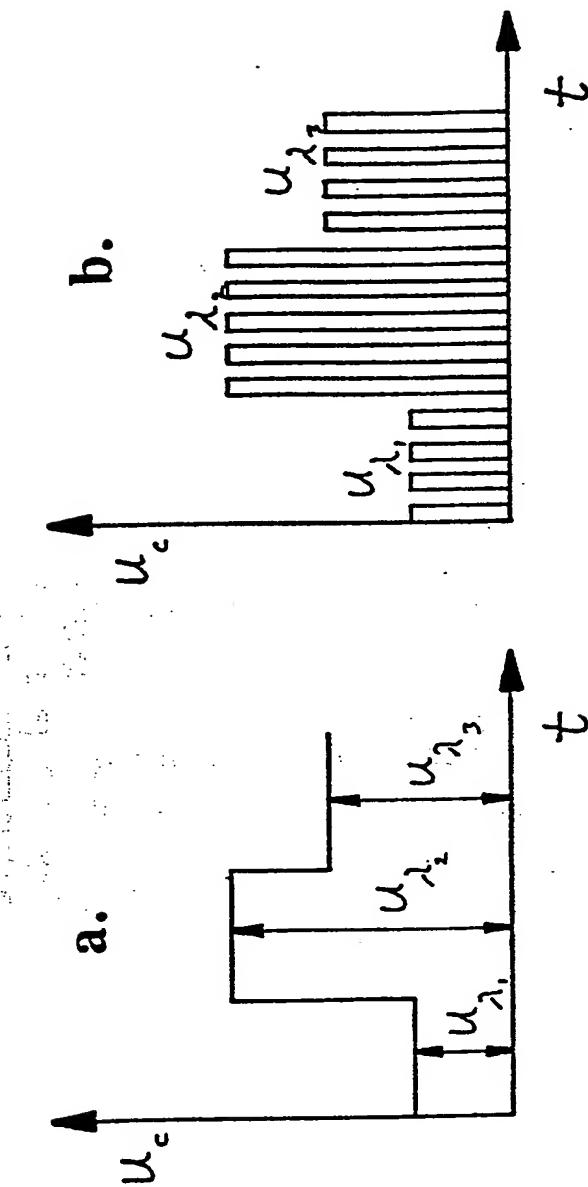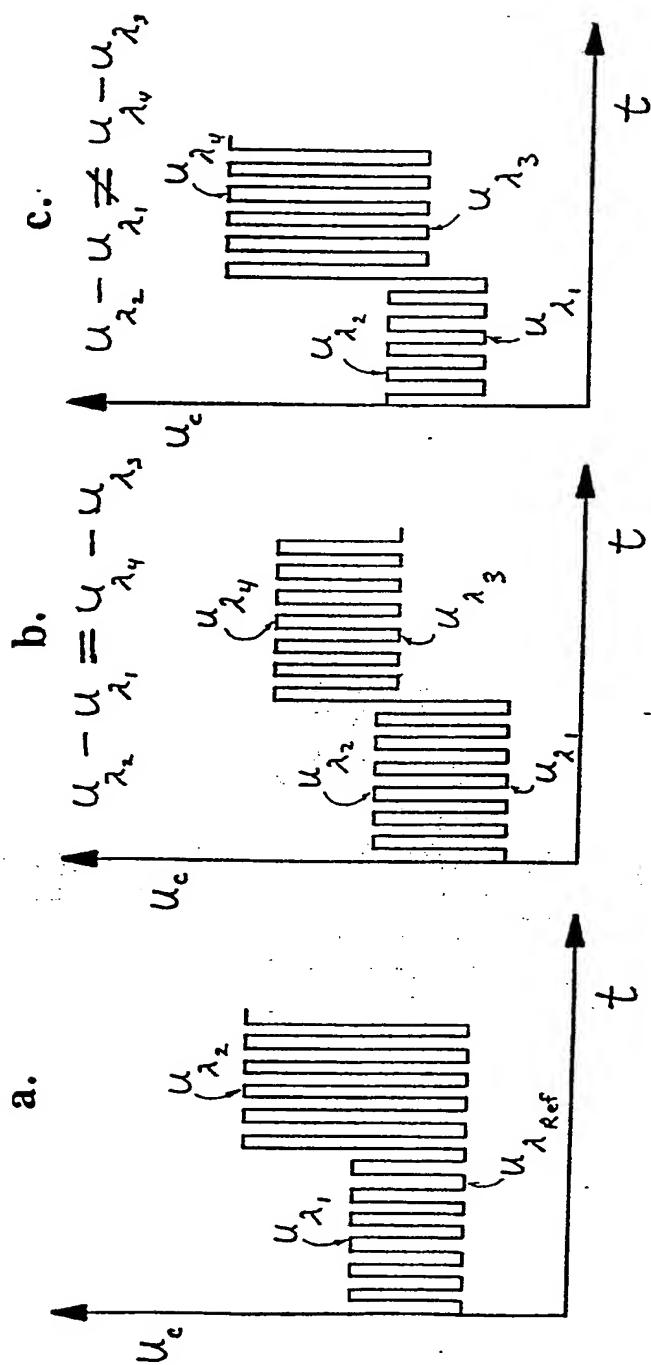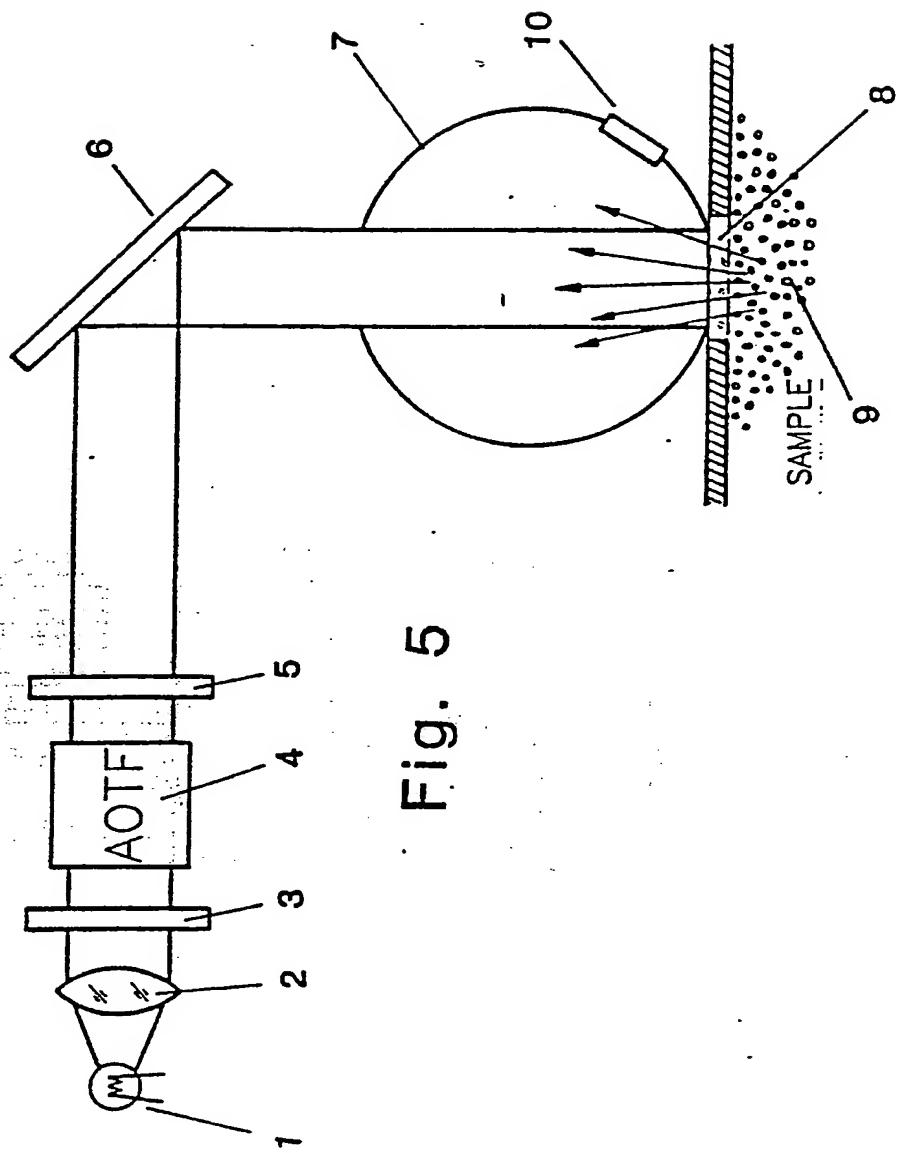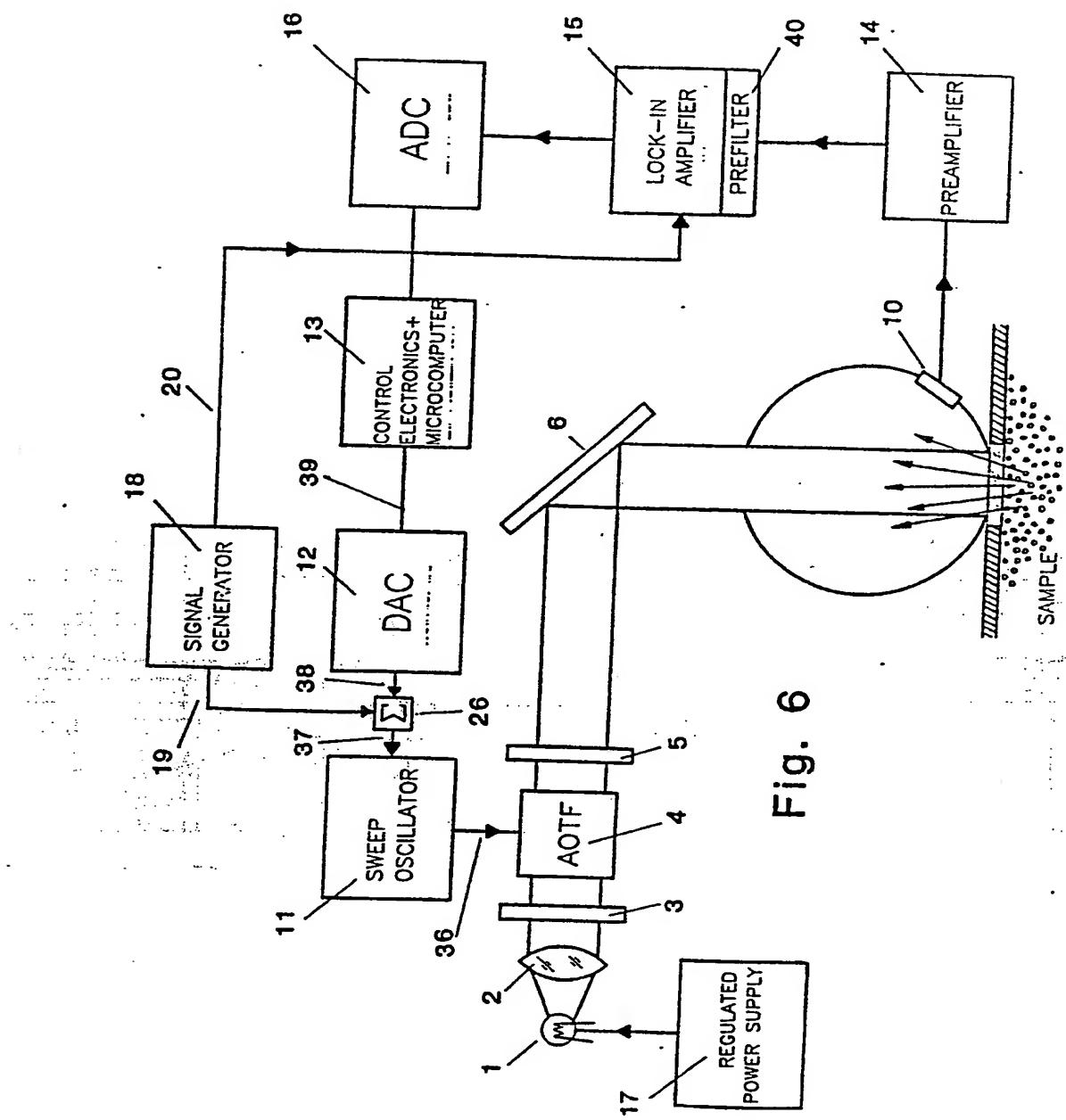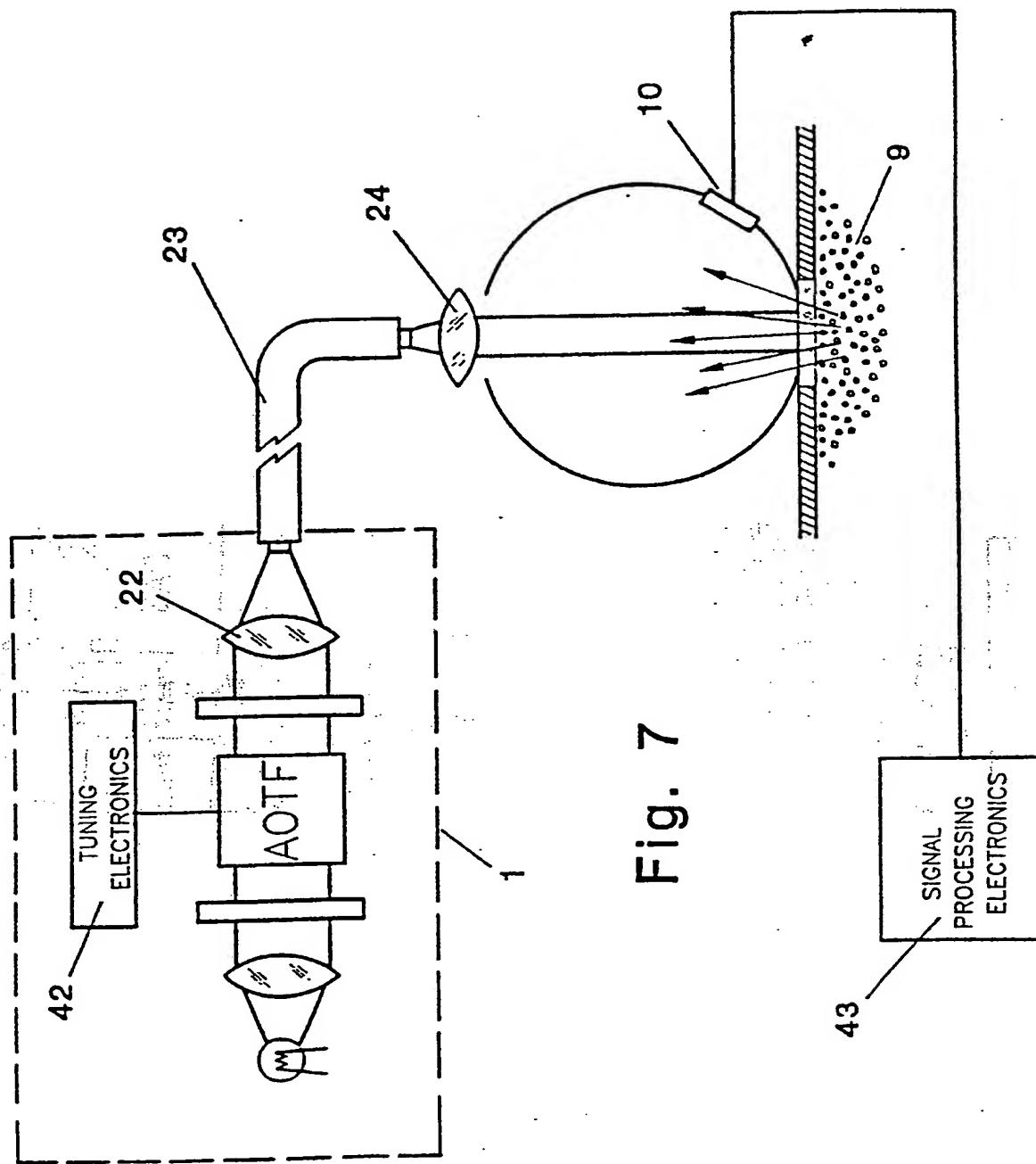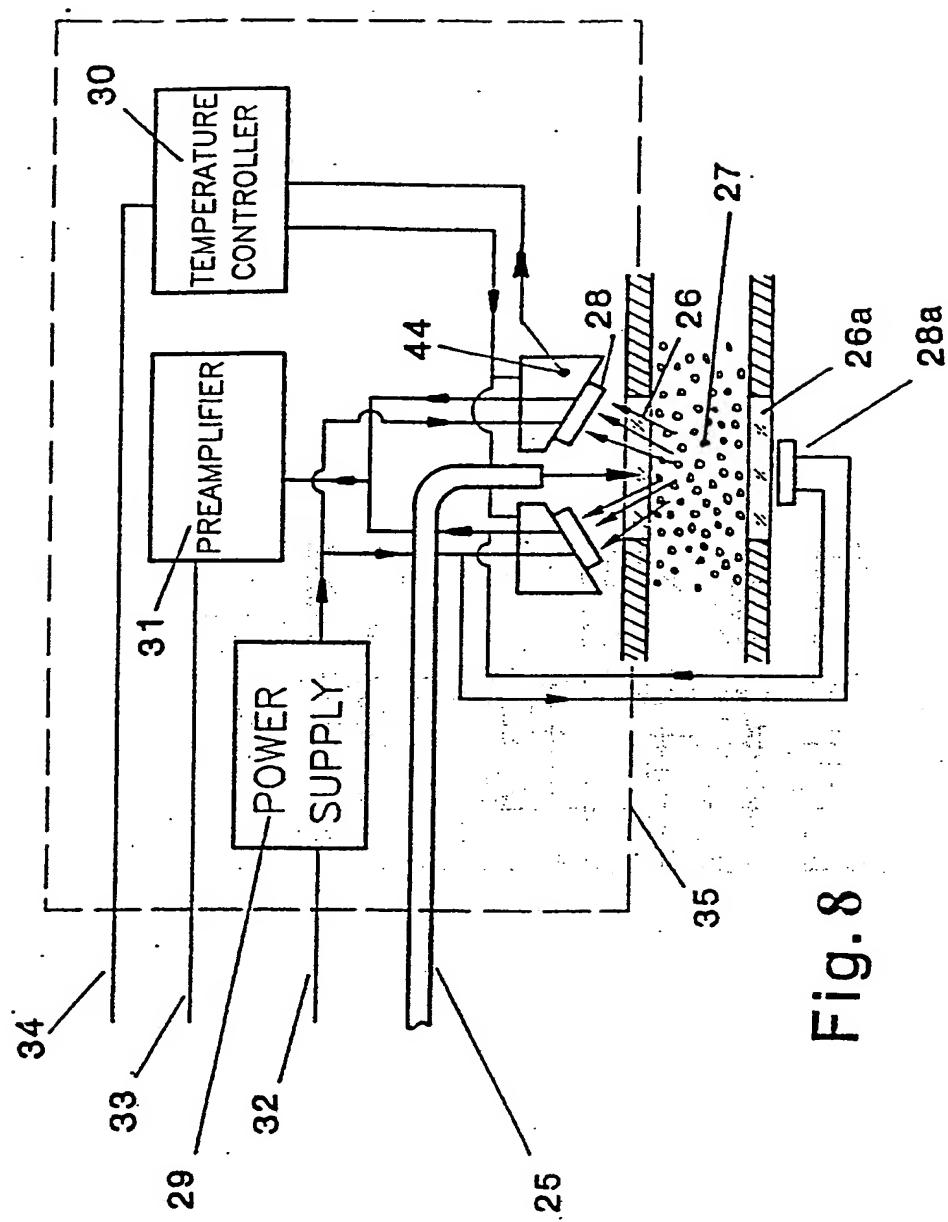
Fig. 8

EUROPEAN SEARCH REPORT

European Patent Office

	DOCUMENTS CONSIDERED TO BE RELEVANT		EP 87303386.4

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.4)
D,A	US - A - 4 285 596 (LANDA) * Abstract * --	1	G 01 N 21/47 G 01 N 21/59 G 01 N 21/35
D,A	US - A - 4 052 121 (I-CHENG CHANG) * Abstract; claims * --	1	
D,A	US - A - 3 828 173 (KNEPLER) * Abstract; column 1 - column 2, line 54 * --	1	
D,A	Analytical Chemistry, vol. 12, Ocotber 1983, American Chemical Society DAVID L. WETZEL "Near Infrared Reflectance Analysis" * Pages 1165A-1176A * --	1	TECHNICAL FIELDS SEARCHED (Int. Cl.4)
D,A	JOURNAL OF THE OPTICAL SOCIETY OF AMERICA, vol. 59, no. 6, June 1969 S.E. HARRIS, R.W. WALLACE "Acousto-Optic Tunable Filter" * Pages 744-747 * --	1	G 01 J 3/00 G 01 N 21/00 G 02 F 1/00 G 06 G 7/00
D,A	APPLIED PHYSICS LETTERS, vol. 25, no. 7, Ocotber 1974 I.C. CHANG "Noncollinear acousto-optic filter with large angular aperture" * Pages 370-373 * ----	1	

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
VIENNA	01-10-1987	ERBER

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
Y : particularly relevant if combined with another document of the same category
A : technological background
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